AD 677118

TECHNICAL REPORT
68-63-CM

DEVELOPMENT OF A LIGHTWEIGHT BUTYL-COATED STRETCH FABRIC

by

James P. Shelley

Rhee Division of Rohm and Haas Company

Warren, R.I.

Contract No. DAAG17-67-C-0009

NOV 18 1968

April 1968

UNITED STATES ARMY
NATICK LABORATORIES
Natick, Massachusetts 01760

Clothing and Organic Materials Laboratory
C&OM-48

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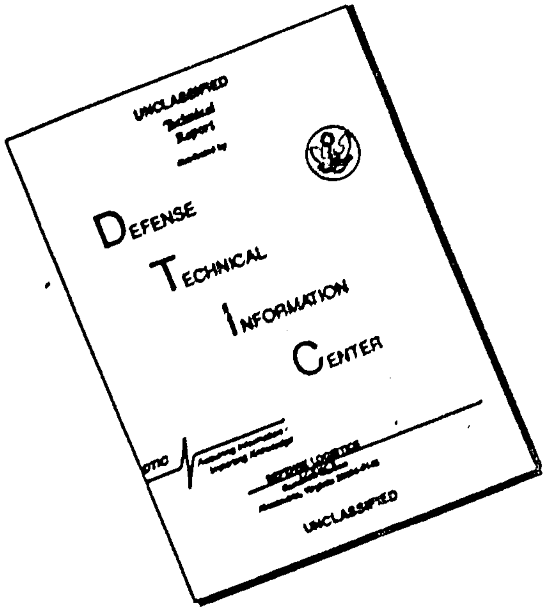

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Project Reference:
1J025601A032

Series: C&OM-48

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FOREWORD

Form-fitting suits for the thermalibrium concept and the EOD (Explosive Ordnance Demolition) protective system are required to reduce the weight and complexity of fabricating techniques and to increase the reliability of the protective systems.

One approach in meeting these requirements is the development of a lightweight, two-way stretch, butyl-coated fabric to be used as one of the layers of the protective clothing. The coated stretch fabric is not to exceed an overall weight of 11.0 ounces per square yard while allowing for the optimum physical and chemical warfare agents protective properties.

Under the guidance of Project Officer Joseph E. Assaf, U. S. Army Natick Laboratories, the processing studies and the development of a technique for the application of butyl compounds to a lightweight, two-way-stretch nylon substrate described in this report were performed by the Rhee Elastic Division of Rohm and Haas Co. through contract number DAAG 17-67-C-009. Chemical warfare agents penetration tests on the butyl-coated fabric were conducted by Edgewood Arsenal, Edgewood, Maryland.

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ABSTRACT

This study was directed toward the development of a technique for the application of multiple thin layers of butyl rubber on a stretch nylon substrate. Since the films of butyl, being of the order of about 1 mil each in thickness, were too thin to calender, a transfer coating technique was considered the best approach, although some consideration was given to the possibility of direct coating. Transfer coating was accomplished by knife-coating a thin film of butyl rubber from a (33% solids) cement on to release paper, drying the film, and then transferring the film from the paper to the fabric by passing film and fabric between doubler rolls. Several problems were encountered with this method but the most serious was extensive shrinkage and loss of warp stretch in the fabric which resulted largely from excessive handling. Several variations in the process designed to minimize fabric handling were explored. The present, and most successful, of these may be described briefly as follows:

Instead of each coat being transferred individually to the fabric, all of the coats are applied to the paper. The fabric is applied on top of the last coat (while the film is still wet) and the entire combination of fabric, film and paper is subjected to the vulcanization process. The release paper is peeled off the coated fabric after vulcanization.

DEVELOPMENT OF A
LIGHTWEIGHT BUTYL-COATED STRETCH FABRIC

1. Introduction

The object of this study was to develop the lightest weight possible in a butyl rubber-coated, two-way stretch fabric where the rubber film is continuous and impermeable to chemical warfare agents and the total weight of the coated fabric does not exceed 11.0 ounce per square yard.

There were three general approaches to the objective. These were (1) calendering, (2) knife-coating from a butyl cement directly on to the fabric and (3) transfer coating. (It is possible, of course, that some combination of these techniques also could be employed).

Considering the nature of butyl rubber, the type of coating desired and prior calendering experience, the calendering method appeared to be the least attractive approach. The only way that a satisfactory coating could be effected by calendering would be by a two-ply technique. This would require considerable experimentation since the heaviest films to be calendered would have a thickness of four mils.

The requirements of this product, it was felt, could best be met by multiple applications of relatively thin coats. For this method the knife-coating techniques (2 and 3 above) are much better suited.

2. Discussion

a. Selection of Fabric

Stretch nylon was considered to be the fabric most suitable for the type of product desired. Based on considerable experience in applying coatings of one kind or another to various types of fabrics, including stretch nylon, it was felt that this fabric offered the best combination of stretch, weave and weight characteristics, although spandex fabrics were also considered.

All of the fabrics used in this study were a Jersey knit stretch nylon made on a circular knitter. The chief criteria for selection of the fabrics were: (1) The weight, (2) the stitch size or tightness of the weave and (3) the stretch qualities. These are summarized in Table I.

TABLE I

FABRICS USED IN STUDY

Style	Yarn	Type Stitch	Weight Oz/yd^2	Stretch[1] Warp	Stretch[1] Fill
#1	Single ply 70 dr. stretch nylon S - Torque Z - Torque	open	3.0 ± 0.3	260%	320%
#2	Same as #1	tight	3.0 ± 0.3	140%	300%
#3	2 ply, 40 dr. stretch nylon	"	2.8 ± 0.3	120%	300%
#4	2 ply, 70 dr. stretch nylon	med.	4.0 ± 0.4	160%	300%
#5	2 ply, 70 dr. stretch nylon	tight	3.0 ± 0.3	90%	300%

b. Butyl Rubber Compositions

Before beginning the study, representatives of the Enjay Division of Humble Oil and Refining Company were contacted for recommendations regarding the type of butyl rubber to be used for maximum resistance to chemical warfare agents and for compounding formulations appropriate for coating applications. Butyl 365 was the type recommended and the formulations examined in the course of this study are shown in Table II.

1 Determined on an Instron

TABLE II

FORMULATIONS

	I	I-B	II	II-B	III	IV
Butyl 365:	100.0	100.0	100.0	100.0	100.0	100.0
Dixie Clay:	120.0	120.0	30.0	30.0	90.0	105.0
Diethylene Glycol:	3.0	3.0	3.0	3.0	3.0	3.0
Zinc Oxide:	10.0	10.0	10.0	10.0	10.0	10.0
Zinc Stearate:	3.0	3.0	3.0	3.0	3.0	3.0
Petrolatum:	5.0	5.0	5.0	5.0	5.0	5.0
Ceresin Wax	5.0	5.0	5.0	5.0	5.0	5.0
Pennac TM-526	1.5	1.5	1.5	1.5	1.5	1.5
MBTS:	2.0	2.0	2.0	2.0	2.0	2.0
Sulfur	1.25	1.25	1.25	1.25	1.25	1.25
Sulfasan R:	1.25	1.25	1.25	1.25	1.25	1.25
531 Yellow Powder:	None	3.0	None	1.50	3.0	3.0
547 Green Powder:	None	1.24	None	0.62	1.24	1.24
507 Red Powder:	None	1.80	None	0.90	1.80	1.80
Pelletex (Black):	None	0.90	None	0.45	0.90	0.90
TiO_2	None	4.00	None	2.00	4.00	4.00
Total	252.00	262.94	162.00	167.57	232.94	247.94

Mixing Cycle for Preparing the Butyl Compositions

Butyl Composition III

Raw Materials	Weight(lb)	Mixing Procedure	Time (Min.)
Enjay Butyl 365	100.00	Break	0
Dixie Clay	90.00	Add Wax	6
Diethylene Glycol	3.00	Add clay, stearate, TiO_2 Glycol & Petrolatum	20
Zinc Oxide	10.00		

TABLE II - Cont'd

Raw Materials	Weight (lb)	Mixing Procedure	Time (Min.)
Zinc Stearate	3.00		
Petrolatum	5.00	Add Thiram, MBTS,)	60
Ceresin Wax	5.00	Sulfur and Sulfasan R)	
Pennax TM-526	1.50		
MBTS	2.00	Add Zinc Oxide	64
Sulfur	1.25		
Sulfasan R	1.25	Cut and Blend	71
531 Yellow Powder	3.00	Add Colors	73
547 Green	1.24		
507 Red	1.80	Cut Out	
Pelletex	0.90		
TiO_2	4.00		
	232.94		

Mill Temp: 66°F
Comments: Went to back roll on break. Mixing continued there. Some sticking to roll on addition of colors. Colors should be masterbatches.

Preparation of the Coating Cement

The solubility of the butyl compound in hydrocarbon solvents, such as toluene and xylene, was readily established by laboratory experiments. A 33% total solids solution in either xylene or toluene provided a suitable combination of solids level and spreadable viscosity. All of the compounding ingredients were first mixed with the butyl polymer using the plant 100-inch, 2 roll mill. The thoroughly mixed compound was then dissolved in the solvent by stirring for approximately 48 hours with a Pony mixer (the cements were never prepared by charging the butyl polymer and compounding ingredients along with the solvent at the Pony mixer).

Preparation of Anchor ("Papi") Coating Cement

To the 100.0 pounds of the regular butyl cement, prepared as described above, was added 3.0 pounds of Upjohn's polyphenyl polymethylene isocyanate ("Papi").

Slab Cures on Butyl Composition III

A series of time/temperature press-curing cycles was run on the rubber mix alone (without fabric) for compositions IB and III to develop a correlation between the time/temperature cycles, and to determine optimum cure and the nature of the cure curve (i.e., whether the properties fall on a plateau and whether or not reversion takes place on overcure). The data, presented below, indicate that while there is a slight drop in tensile as the cure becomes tighter, the other properties (modulus and elongation) tend to level off fairly well and no actual reversion takes place. In short, both compositions are considered as relatively flat curing. (See Table III)

TABLE III

PRESS CURING OF COMPOSITIONS III AND IB

Curing Conditions		Gage	Modulus(psi)		Tensile Strgth.	Elong.	Break Set
Time (Min.)	Temp. (°F)	Inches	300%	500%	psi	%	%
		BUTYL COMPOSITION III					
120	245	0.0263	275	545	1920	810	115
120	245	0.0257	289	590	1940	820	115
180	245	0.0255	345	800	1880	740	110
180	245	0.0252	335	790	1850	720	105
240	245	0.0282	355	890	1780	690	110
240	245	0.0292	355	900	1740	670	100
120	260	0.0282	340	830	1880	700	112
120	260	0.0271	350	900	1950	710	113
180	260	0.0271	415	1080	1680	600	96
180	260	0.0268	420	1100	1700	600	98
240	260	0.0301	450	1250	1540	570	91
240	260	0.0305	460	1240	1660	590	96
20	320	0.0241	405	1080	1800	620	100
20	320	0.0260	405	1100	1850	640	102
30	320	0.0236	440	1280	1620	560	85
30	320	0.0246	440	1280	1650	570	90
50	320	0.0293	440	1240	1620	550	81
50	320	0.0298	440	1300	1640	560	81
		BUTYL COMPOSITION IB					
120	260	0.0287	390	950	1730	680	130
120	260	0.0291	390	910	1560	660	120
180	260	0.0272	455	1180	1560	600	115
180	260	0.0258	450	1120	1620	610	115
240	260	0.0272	485	1160	1540	580	105
240	260	0.0276	490	1200	1520	580	106

Calendering of Butyl Rubber

The only experiment done with calendering of butyl was based on a highly loaded composition[2] and was run on a laboratory scale calender. It is generally felt that finer quality calendering is more obtainable with this apparatus than on the production size unit (hence, even success at this level is no assurance that the composition is suitable for large scale calendering). In this run we were unable to obtain a continuous sheet any finer than about 20 mils—due largely to the inherent softness of this particular formulation. Ordinarily we would expect to be able to calender sheets as fine as 4-5 mils thick. It was felt at this point that a wide variety of compositions would have to be explored and possibly much tighter temperature control maintained before any hope of obtaining a substantially thinner continuous sheet could be entertained.

c. Variables Studied in the Transfer Process

At the outset of this work the following variables were seen as those requiring most immediate definition:

1. Choice of fabric type
2. Optimum (total) coating weight of the fabric.
3. Optimum film thickness for each coat.
4. Drying conditions (i.e., oven temp. vs. dwell time).
5. Whether or not an "anchor coat" would be required.
6. Most suitable solvent for the cement.
7. Effect of loading (clay) level in the butyl composition.

(1) Fabric Study

In the initial work with the transfer process, the fabric used was a lightweight (ca. 3.0 ± 0.3 oz/yd^2), loosely woven stretch nylon having excellent stretch characteristics in both warp and fill directions. This fabric was designated as Style #1. Regardless of the measures employed to minimize the tendency, the coated Style #1 invariably exhibited numerous "pin holes" throughout the entire length and breadth of the samples. Running data for the variables studied in the coating process are presented in Table IV (Runs 1 through 21). Variations in the technique of applying the film to the fabric were studied in Runs 23 through 28 (Table VI). It soon became obvious that Style #1 was not suitable for the requirements of this product and attention was then shifted to Style #4. This fabric lent itself much more readily to coating with butyl film as evidenced by the better film continuity and absence of "pin holes" in the vulcanized product. At the same time, however, it was a 25% heavier weight fabric and this, it was felt, was highly undesirable and should be avoided. Furthermore, it was not the weight of the Style #4 fabric per se that made it more adaptable to coating but the fact that a "tighter" stitch was employed. Following a discussion of this problem, several experimental fabrics were submitted for testing. These were Styles #2, 3 and 5. (See Page 2 for a description of these fabrics). In the coating

2. Designated as Butyl Composition I.

runs that followed, it became apparent that Style #5 was unsatisfactory because of the inherently poor stretch qualities, although it coated satisfactorily. Style #3 was judged undesirable for further study because it was somewhat similar to Style #1 in handling characteristics, being too easily stretched and subject to "pin hole" formation. The stretch characteristics of Style #2, after coating, left something to be desired, but it was felt that this was the result of improper handling of the fabric and could be eliminated or minimized with further study. Except for this deficiency, Style #2 was considered to provide the best all-round combination of properties for use as the base fabric and all runs from No. 21 on were conducted with this fabric.

(2) Optimum Coating Weight

One way of defining optimum coating weight would be: lightest weight coating that will still provide satisfactory protection as indicated by resistance to penetration by chemical warfare agents. The results obtained in the "100-yard" sample runs (Tables VII and VIII) by simple inspection, however, indicated that the lightest weight fabric (ca. 8 oz/yd^2 total weight) definitely would not afford adequate protection against chemical warfare agents. Close inspection of the fabric over a lighted table showed numerous "pin holes" throughout the fabric. The medium weight (ca. 10 oz/yd^2 total weight) fabric was "border line" showing occasional pin holes. The heavy (ca. 11 oz/yd^2 total weight) fabric had relatively few pin holes (which were marked during inspection) and, on this basis, was selected for application to the 500-yard sample.

(3) Optimum Thickness for Each Coat

Film thickness studies showed that a uniform film of butyl as thin as 0.5 mils (dry) could be applied and readily transferred. It was noted, however, that prolonged running at this thickness tended to give rise to "bare streak" defects in the film which, in turn, apparently resulted from butyl "skin" snagging on the knife blade at the low setting. A certain amount of this type of occurrence seems to occur at almost any knife setting but does tend to become more frequent as the knife setting goes down. The objective, in general, was to lay the films as thin as possible--usually about 1/2 to 1 mil each to provide the maximum number of coats for any one weight level. Note that in Run 29 (Table VII) a total of four coats gave a product averaging about 9.55 ounce per square yard, while in Run #30 the same number of coats gave only 7.6 oz/yard^2 because most of the coats were thinner.

(4) Drying Conditions

The importance of driving off all of the solvent from the butyl film was never underrated. The only question was the criterion to employ as a means of characterizing the films in this regard. In the early work (Runs 3, 4 and 5, Table IV), samples of the dried film were tested on a moisture balance and the results indicated that with three units operating at 150°F the films were free of residual solvent.

(5) Anchor Coating

From previous experience with other rubber-coated fabric systems, it was thought that an "anchor coating" might at least be desirable, if not actually mandatory, for good film-to-fabric adhesion. Good adhesion is generally obtainable through the addition of a polyfunctional isocyanate "Papi" to the cement and it was felt that a similar treatment could be employed in the butyl system if experience so indicated. Adhesion data (Table V) show that without Papi the adhesion was rather erratic, varying from 4.5 lbs/2-inch to "infinite" with many of the samples being in the "too low" range, namely, less than 8.0 lbs/2-inch. The addition of Papi (samples 1 and 9 of run No. 20-Table VI) brought the adhesion level to the range of 8-11 pounds per 2 inches of width which was considered adequate.

(6) Most Suitable Solvent for the Cement

Xylene is a slight preference over toluene as a solvent for the butyl cement. Xylene is less volatile but toluene cements were noted to have a tendency to "skin over" which led to defects in the film when the skin would snag on the knife blade.

(7) Effect of Loading (Clay) Level in the Butyl Compound

A low level of loading (i.e., about 30 parts clay per 100 parts butyl polymer (Butyl Comp II)) gave a soft and still slightly tacky film even after curing. This type of composition tended to adhere to the silicone paper used for interleaving[3] and resulted in a film having a relatively rough surface. A clay loading in the range 90-105 parts gives a considerably more uniform surface. Butyl Comp. III (90 parts clay) appears to be about optimum; 105 parts clay (Butyl Comp. IV) resulted in reduced "pick up" by the fabric in the transfer operation.

3 Test Methods

a. Adhesion

For the first 19 runs (i.e., the first 6 months work), the following test procedure was used:

1-inch x 6-inch strips were die-cut from a sample of the product and a small (ca. 1/4") length of the film peeled from the fabric. Film and fabric were then clamped in separate jaws of an Instron testing machine and the pounds pull required for delamination of the strip recorded with the machine pulling at the rate of two inches per minute. Adhesion values were expressed in pounds per inch of width and the average of five determinations was recorded as the adhesion.

[3]The coated fabric was wrapped around a cylindrical cardboard core with Carter Rice's 60 lb. silicone paper as an interleaving release paper in order to prevent adhesion of the butyl film to the reverse side of the fabric during the curing process.

For the remainder of the study (runs 20 through 37), the adhesion was determined in accordance with Test Method No. 5970 outlined in Federal Specification CCC-T-1916, entitled Textile Test Methods - dated 15 May 1951.

b. Overall Weight

Test Method No. 5041 of Federal Specification CCC-T-1916, dated 15 May 1951 and entitled Textile Test Methods, was used for all weight determinations.

c. Width

Test Method No. 5020 of Federal Specification CCC-T-1916, dated 15 May 1951 and entitled Textile Test Methods, was used for all width determinations.

d. Breaking Strength and Elongation at Break

All samples were tested in accordance with Test Method No. 5102 of Federal Specification CCC-T-1916, dated 15 May 1951, entitled Textile Test Methods.

e. Tear Strength

All samples were tested in accordance with Test Method No. 5134 of Federal Specification CCC-T-1916, dated 15 May 1951, entitled Textile Test Methods.

f. Modulus at 50% Elongation

The following method was used: A test specimen 2 x 3 inches was inscribed with a 1-inch gage length and tested on an Instron at a speed of 5 inches per minute. The specimen was stretched to 75% and then immediately relaxed. The sample was stretched again and the number of pounds recorded when the elongation reached 50% (or when the one-inch gage marks were 1 1/2-inches apart).

g. Low Temperature Resistance

For Runs 1 through 19, samples were tested in accordance with Test Method No. 5874 of Federal Specification CCC-T-1916, dated 15 May 1951, and entitled Textile Test Methods. For Runs 20 through 32, an 8 x 8" sample was placed in a cold box for 30 minutes, then (while still in the box) folded over with the coated side up and rolled with a 10-pound roller, and examined for cracks or other signs of failure. If none were visible, the temperature of the box was lowered another 3-5°C and the test repeated until some failure occurred. The failure temperature and the immediately preceding passing temperature were recorded.

h. Hydrostatic Resistance

All samples from Runs 20 through 32 were tested in accordance with Test Method No 5512 of Federal Specification CCC-T-191b, dated 15 May 1951 (Textile Test Methods) A Mullen tester was used Runs 1 through 19 were not tested for hydrostatic resistance

i After-Strength of Coating

Runs 1 through 19 were not tested for after-strength of the coating. Runs 20 through 32 were tested in accordance with the procedures described in Test Method No 5972 of Federal Specification CCC-T-191b, dated 15 May 1951, and entitled Textile Test Methods.

j Resistance to CW Agents Penetration

The chemical warfare agents penetration tests were conducted by Edgewood Arsenal in accordance with the following test methods outlined in MIL-STD-282, dated 28 March 1956: Filter Units, Protective Clothing, Gas-Mask Components and Related Products: Performance Test Methods:

Mustard - Method 204.1 1 - Mustard Resistance of Impermeable Materials (Static-Diffusion Method)

GB - Method 206.1 1 - GB-Vapor Resistance of Impermeable Materials (Static-Diffusion Method)

4. Experimental Procedure

For Runs 1 through 22, the butyl film was knife-coated on to Stripkote AR buff/white, a one-side release paper, from a 33% solids cement and immediately passed into the drying over at a rate of about 10 feet per minute On emerging from the oven the film passed for about 5 yards over idler rolls to the doubler unit. Except for Run 22, the fabric was applied to the dry film in the area between the dry end of the oven and the doubler unit Fabric, butyl film and paper then passed through the bite of the doubler rolls (the top roll of which was heated to about 175°F) at a pressure usually of 80 pounds per lineal inch--whereupon the softened butyl film would adhere to the fabric The butyl-coated fabric would emerge from the bite and be wound up on one roll and the paper, now essentially free of any butyl film, wound up on another roll. This process was repeated for each and every coat applied to the fabric for Runs 1 through 21 In Run 22, the fabric was laid on the wet film at the "wet" end of the oven-for the first coat only-and traveled the length of the oven along with the film to obtain more intimate contact between the butyl film and fabric All other coats were then applied by the transfer process as described above This procedure was abandoned because of occasional difficulties encountered in obtaining satisfactory release of the film from the paper Significant shrinkage and loss of stretch with coated fabric also occurred.

Run 23

For the first coat, the butyl cement (containing Papi) was knife-coated on to a 60-lb. silicone duplex release paper. The fabric was laid on the wet film at a point just beyond the knife paper. Film and fabric then passed together through the drying oven at about 10 feet per minute. On emerging from the oven, the fabric, film and paper passed between the doubler rolls (upper roll steam heated to 175^{o}F, lower roll unheated) where the butyl-coated fabric was parted from the paper. Both paper and coated fabric were wound on separate cores.

For the second, and all subsequent coats, the butyl film was applied to the paper and dried in the same manner as the first coat. The fabric, however, was not brought into contact with the film until after it emerged (dry) from the oven. At a point just before entering the doubler, the fabric was laid against the film and then the film, fabric and paper again underwent the transfer operation in the same manner as the first coat. Extensive shrinkage and loss of stretch in the product continued to occur during the coating operation. It was concluded that some means of either minimizing handling of the fabric or of securing it so that it would resist shrinking would have to be introduced. The latter technique was investigated in Run No. 24.

Run 24

In this run, canvas duck coated with uncured rubber was employed as a carrier or anchor fabric in an effort to prevent or minimize shrinkage of the Style #2 fabric during the transfer coating operation. (This is a technique which has been successfully employed in other coating operations where the objective is to apply a coating to a stretch fabric without taking any of the stretch out of the fabric). Style #2 (10-yard length, 18" wide) was interleaved under tight wrapping conditions on the canvas duck. This operation combined with the "tacky" character of the rubber film was sufficient to provide a reasonably tight bond of Style #2 to duck. A bond that is too tight is not desirable as it tends to cause loss of stretch in the coated fabric when it is stripped off the carrier cloth.

The Style #2 fabric was coated in the same manner as in Run 23 with the exception, of course, that the fabric in this case was bonded to another fabric. Both fabrics went through the oven together and it was noted that once inside the oven the Style #2 came unbonded from the carrier fabric. This, of course, completely nullified the effect of the carrier cloth and the usual fabric shrinkage occurred. In addition, considerable handling problems occurred during the doubling operation because of the presence of the heavy canvas duck and numerous creases were introduced. After two passes through the oven, the run was aborted and the "carrier cloth" approach was abandoned.

Runs 25 through 28

The 60-pound silicone (duplex release) paper was coated and the film dried to give a 1-mil dry film on paper. The butyl-coated paper was then rolled up, brought back to the feed end of the unit and a second 1-mil coat applied and dried over the first coat. This operation was repeated until one less than the number of coats desired was on the paper. The last coat was a Papi-containing coat and the fabric (Style #2) was laid on top of the wet film and both film and fabric went through the oven together. After emerging from the oven, the fabric, film and paper were passed between the doubler rolls where the transfer operation was carried out in the usual manner. The coated fabric was wound on a cardboard core and cured 90 minutes at 300°F in a circulating air oven, then dusted with Italian Talc No. EGT EXTRA 00000.[4]

The concept of applying all the butyl coats to the paper and then transferring the entire coating, in one operation, to the fabric had been considered much earlier in the study--at the time when the Stripkot paper was being used. The heavy character of the paper, however, made roll-up without creasing virtually impossible and this approach was temporarily shelved until experimentation with the lighter weight silicone paper was begun (Run 23).

Runs 29 through 37

The 60 pound silicone (duplex release) paper was coated and the film dried to give 0.5 to about 1.25 mil film on paper. The butyl-coated paper was then rolled up, brought back to the feed end of the unit and a second 0.5 to 1.25 mil coat applied and dried over the first coat. This operation was repeated until one less than the number of coats desired was on the paper. The final coat was a Papi-containing coat and the fabric (Style #2) was laid on top of the wet film and both film and fabric went through the oven together. After emerging from the oven, the fabric, film and paper were wound on the vulcanizer drum (the diameter of which is 40.0 inches) without previously being subjected to a transfer operation as in Runs 25 through 28. The drum was then placed in a radio frequency vulcanizer and vulcanization was carried out at 300°F for 90 minutes under 30-pound nitrogen pressure. The 90-minute vulcanization time was measured from the time at which the temperature of the mass actually reached 300°F. The temperature at the start of the vulcanization was about room temperature or slightly above and the "rise time" varied from 45 minutes to one hour, so that the total time in the vulcanizer for the samples was about 2-1/4 to 1-1/2 hours. After removing the drum from the vulcanizer, the silicone release paper was peeled from the rubber surface of the fabric and the surface of the rubber was dusted with No. EGT EXTRA 00000 Italian Talc.

[4] Supplied by Whitaker, Clark and Daniels.

5. Equipment

a. Transfer Coating Equipment

(1) The coater is a knife over roll type. The knife itself is constructed so that the bow can be varied in order to accommodate different width coatings (compensates for edge effects due to flow of the wet film).

(2) The drying oven is steam-heated, parallel air flow having four temperature zones, all of equal length. The overall length of the oven is 150 feet and the belt (paper) speed can be varied between 0 and 50 ft/min.

(3) The doubler unit is basically two rolls, each 80 inches long. The top roll is a heated steel roll, 12 inches in diameter. The bottom roll is a hard rubber roll (85 Durometer) which usually is not heated. The roll pressure is variable between 0 and 80 pounds per lineal inch of nip.

b. Curing Equipment and Procedures Followed

Details on the construction and operation of the radio frequency vulcanizers in use at the Rhee Division may be obtained by consulting U.S. Patents 2,703,436 and 2,743,479. The chief advantage of radio frequency over the conventional steam cure is that a considerably greater mass of rubber can be cured with a higher degree of uniformity. The high-frequency electric field penetrates the entire mass of rubber and raises the temperature thereof uniformly. Around the outside of the rubber the air temperature is brought up at the same rate as the mass temperature through the use of steam coils, so that at any one time within the vulcanizer there are no large temperature differentials that might result in over-curing some areas and under-curing others.

The rubber/fabric laminate is wrapped on specially constructed drums (silicone paper is generally used as an interleaver) which rotate slowly within the vulcanizer during the curing period.

The small samples (5 yards or under) run in this study were cured in a circulating air oven (in the laboratory) because they were much too small to be cured via R. F. equipment. The samples were wrapped around a cardboard core 3-1/2 inches in diameter with 60-pound silicone paper as interleaver, and heated in a circulating air oven for 90 minutes at 320°F. Except for the uniform quality of the cure, where large quantities of rubber are involved, there is essentially no difference between the characteristics of butyl rubber cured in a hot air oven and that cured in the R. F. vulcanizer. Runs 17, 23, 27, 29, 30, 31, and 32 through 37 were cured in the R. F. vulcanizer - all others in a circulating air oven.

6 Summary

Various techniques for effective use of the transfer method of applying thin films of butyl rubber to fabric were explored. These included:

a. Knife-coating a thin film of butyl rubber from a solvent cement on to release paper, drying the film, and then transferring the film to the fabric by laying the fabric on top of the dry film and passing film and fabric between heated "doubler" rolls. This process was repeated as many times as the number of films applied to the fabric.

b. Technique similar to (a) above except that for the first pass only the fabric was applied to the film while the latter was still wet Fabric and film traveled through the drying oven together and then passed through the doubler unit where the film transferred to the fabric. All subsequent coats were applied by the method described in (a) above.

c. Use of (uncured) gum rubber-coated canvas as a carrier fabric to provide a semi-rigid substrate for the fabric to be coated. This was tried as a means of minimizing shrinkage and creases frequently introduced in handling the fabric. The coating process itself was otherwise similar to (b) above.

d. Thin (about 1/2 to 1.0 mil dry) films of the butyl composition were successively applied to the release paper until one film less than the total coating level desired on the fabric was on the paper. With the application of the final coating, the fabric was also applied to the wet film. Film and fabric were then dried together and passed through the doubler unit where the film transferred to the fabric. This process gave far better results as regards shrinkage and creasing of the fabric than any of the previous processes. After transfer, the fabric was wrapped on a drum and cured in the R. F. vulcanizer.

e. Thin (ca. 1/2 to 1.0 mil dry) films of the butyl composition were successively applied to the release paper until one film less than the total coating level desired on the fabric was on the paper. With the application of the final coating, the fabric was also applied to the wet film. Film and fabric were then dried together as in the process described in (d) above, but on emerging from the oven there was no transfer operation. Instead, fabric, film and paper were wound on the curing drum and the composition cured in the R. F. vulcanizer. The silicone release paper was removed from the coated fabric after the curing operation.

7. Conclusions

The objective of this study appears to have been attained with the process as exemplified by Runs 29 through 37. In principle, certainly, the process is applicable and no great obstacle to its practical application is apparent at this time. Experience to date with the process has, however, been limited only to these five runs, and the presence of a few "pin holes" in isolated portions of the fabric suggests that perhaps an additional drying step for the butyl films may be desirable. Other modifications of the process may, of course, also be indicated as more running experience is accumulated, but the overall picture as regards the ability to produce large (viz. 10,000 yards) quantities of the product is a very optimistic one - there is no significant deterrent at the present time.

8. Acknowledgments

The author wishes to acknowledge the generous help and suggestions provided by Mr. Joseph E. Assaf of the U.S. Army Natick Laboratories.

Acknowledgment is also made to the Enjay Division of Humble Oil and Refining Company for the extensive literature and helpful suggestions for the compounding and processing of butyl rubber.

TABLE IV

DATA ON COATING RUNS 1 THROUGH 22

Run No.	Butyl Comp. No.	Cement Comp.	Fabric	Coats Applied	Drying Temperatures °F First Unit	Second Unit	Third Unit	Fourth Unit	Wt. of Uncoated Fabric (oz/yd^2)	Wt. of Coated Fabric (oz/yd^2)	Notes: Samples coated were 5-7 yds. in length and about 30 in. wide except where noted otherwise.
1.	II	33% in Toluol	Style #1	(1) 3 mil	R.T.[1]	R.T.	R.T.	R.T.	3.3	6.9	Dwell time in oven=15 minutes for all film. Good film. No evidence of blistering but numerous holes readily visible on stretching (after curing).
2.	II	"	"	(1) 5 mil	R.T.	170	190	R.T.	3.3	9.2	Film has considerable blisters.
				"	R.T.	150	150	R.T.	"	-	Film has a few blisters.
				"	R.T.	R.T.	R.T.	R.T.	"	-	Very slight evidence of trapped solvent in film.
3.	II	"	"	(1) 3 mil	150	170	170	R.T.	"	-	Some blisters in film. Volatiles content of film is nil.
	II	"	"	"	150	150	150	R.T.	"	6.7	A few blisters in film. Volatiles content nil.
	II	"	"	"	R.T.	"	"	R.T.	"	-	No blisters in film. Volatiles content nil.
	II	"	"	"	R.T.	R.T.	150	R.T.	"	-	No blisters in film. Volatiles: 1.7%.
4.	II	33% in Xylene		(1) 3 mil	R.T.	R.T.	150	R.T.	"		No blisters in film. Volatiles: 2.2%.
	II	"	"	"	R.T.	150	150	"	"	-	No blisters in film. Volatiles: nil.
	II	33% in Xylene	Style #1	(1) 3 mil	150	150	150	R.T.	3.3	-	No blisters in film. Volatiles: nil.
	II	"	"	"	150	175	175	"	"	6.2	No blisters in film. Volatiles: nil. Some stretch apparently taken out of fabric.

TABLE IV
(Cont'd)
DATA ON COATING RUNS 1 THROUGH 22

Run No.	Butyl Comp. No.	Cement Comp.	Fabric	Coats Applied	Drying Temperatures °F First Unit	Drying Temperatures °F Second Unit	Drying Temperatures °F Third Unit	Drying Temperatures °F Fourth Unit	Wt. of Uncoated Fabric (oz/yd²)	Wt. of Coated Fabric (oz.yd²)	Notes: Samples coated were 5-7 yds. in lgth. and about 30 in. wide except where noted otherwise.
5.	II	33% in Xylene	Style #1	3/3[2]	150	150	150	R.T.	3.3	10.7	No evidence of blisters in any film. Adhesion appears excellent but on stretching all samples showed numerous pin holes. Volatiles-nil.
	II	"	"	3/2	"	"	"	"	"	9.9	
	II	"	"	3/2/1	"	"	"	"	"	11.0	
	II	"	"	3/1/1	"	"	"	"	"	10.3	
6.	IIB	33% in Xylene	"	3/1/1/1	150	150	150	R.T.	3.2	11.5	All films show good continuity but still show pin holes on stretching.
7.	IIB	"	"	2/2/1/1	"	"	"	"	"	10.6	
8.	IIB	"	"	2/1/1/1	"	"	"	"	"	9.9	About 50% of the stretch has been taken out of the fabric.
9.	IIB	"	"	1/1/1/1	"	"	"	"	"	9.0	
10.	IIB	"	"	2/2/1	"	"	"	"	"	9.6	Same as above though only about 25% of the stretch has been removed.
11.	IIB	33% in Xylene	Style #4	2/2/1	"	"	"	"	4.0	[illegible]	Good coating. No pin holes on stretching.
12.	IIB	"	Style #2	2/2/1	150	150	150	R.T.	3.1	9.3	Good coating. No pin holes on stretching.
			Style #2	1/1/1	"	"	"	"	3.1	7.5	Good coating but adhesion is less than 1.0 lb/in-probably because of too light a base coat.
13.			Style #5	1/1/1/1	"	"	"	"	"	8.1	
			"	1/1/1	"	"	"	"	3.2	7.[illegible]	
			"	1/1/1/1	"	"	"	"	"	8.1	

TABLE IV
(Cont'd)
DATA ON COATING RUNS 1 THROUGH 22

Run No.	Butyl Comp. No.	Cement Comp.	Fabric	Coats Applied	Drying Temperatures °F First Unit	Second Unit	Third Unit	Fourth Unit	Wt. of Uncoated Fabric (oz/yd^2)	Wt. of Coated Fabric (oz.yd^2)	Notes: Samples coated were 5-7 yds. in lgth. and about 30 in. wide except where noted otherwise.
14.	III	33% in Xylene	Style #5	3/1/1	"	"	"	"	"	9.9	
				3/1/1/1	"	"	"	"	"	10.3	
15.	IV	33% in Xylene	Style #4	3/1/1	"	"	"	"	4.0	9.8	Higher level of clay in coating comp.-no significant change in properties or quality of the product.
				3/1/1/1	"	"	"	"	"	11.0	
16.	III	"	Style #4	3/3/1	150	150	150	R.T.	3.9	11.8	All films show good continuity and no pin holes on stretching.
		"	"	3/2/2	"	"	"	"	"	11.7	
			"	3/3	"	"	"	"	"	10.7	
			"	3/2	"	"	"	"	"	9.3	Two samples over weight limit.
			"	3/1/1	"	"	"	"	"	9.5	
17.	III	"	"	3/2	"	"	"	"	3.9	9.4	50 yard run made to check R.F. cure vs. oven cure.
18.	III	33% in Xylene	Style #5	3/2	150	150	150	R.T.	3.2	9.2	
			"	3/2/2	"	"	"	"	"	11.2	
			"	3/2/1	"	"	"	"	"	10.4	
			"	3/1/1	"	"	"	"	"	9.0	
				2/2	"	"	"	"	"	8.3	
			"	2/1/1	"	"	"	"	"	8.6	
			Style #2	3/2/1	"	"	"	"	3.0	9.8	
19.	III	"	Style #4	2/2/2	150	150	150	R.T.	4.3	12.3	
			"	2/2/1	"	"	"	"	"	11.0	
			"	2/1/1	"	"	"	"	"	10.0	
			"	3/2/1	"	"	"	"	"	12.1	
			"	3/1/1	"	"	"	"	"	11.1	
			Style #1	2/2/1	"	"	"	"	[illegible]	9.1	
			"	3/2/1	"	"	"	"	"	10.6	
			"	3/1/1	"	"	"	"	"	[illegible]	
			Style #3	2/1/1	"	"	"	"	2.75	9.8	
			"	3/1/1	"	"	"	"	"	9.6	

TABLE IV
(Cont'd)
DATA ON COATING RUNS 1 THROUGH 22

Run No.	Butyl Comp. No.	Cement Comp.	Fabric	Coats Applied	Drying Temperatures °F First Unit	Second Unit	Third Unit	Fourth Unit	Wt. of Uncoated Fabric (oz/yd²)	Wt. of Coated Fabric (oz.yd²)	Notes: Samples coated were 5-7 yds. in lgth. and about 30 in. wide except where noted otherwise.
20.	III	33% in Xylene	Style #2	2/1/1/1	150	150	150	R.T.	3.0±0.3	10.22	Samples post-dried 15'/250°F, then cured 2 hrs. 15 min. at 300°F in a circulating air oven. Runs 1, 2, 3, 4 and 9 had PAPI added to the first coat. Samples 2,3,4, and 5 were subsequently rejected as containing too many creases.
			"	2/1/1	"	"	"	"	"	7.43	
			"	1/1/1/1	"	"	"	"	"	8.34	
			"	1/1/1	"	"	"	"	"	7.83	
			"	2/1/1/1	"	"	"	"	"	[illegible]	
			"	2/1/1	"	"	"	"	"	[illegible]	
			"	1/1/1/1	"	"	"	"	"	9.2[illegible]	
			"	2/1/1	"	"	"	"	"	8.21	
	III	"	Style #1	2/1/1/1	150	150	150	R.T.	3.0±0.3	8.74	
21.	III	"	Style #2	2/1/1	"	"	"	"	"	-	Coated three 80 yd. lengths 53" wide for each sample. All samples post-dried 15'/[illegible], then cured [illegible] at 300°F in an RF vulcanizer. PAPI was added in the first coat of all samples. "Wet" application of the base coat (no PAPI added) followed by transfer application of three more coats.
				2/1/1/1/1	"	"	"	"	"		
			Samples from Run 21 were not tested for Physical Properties because of severe shrinkage during processing and numerous defects present.								
22.	III	"	Style #2	1/1/1/1	150	150	150	R.T.	"	-	
			Run 22 was not tested for Physical Properties-this was a small piece of fabric and only qualitative observations were in order.								

Note: 1. Denotes "Room Temperature".

2. Each figure represents the [illegible] of the coating [illegible]. The number of figures represents the number of coats. For example [illegible] would be interpreted to mean a three-coat application, the first coat being [illegible] mils thick, the second [illegible] thick, and the third 1 mil thick.

TABLE V

PHYSICAL CHARACTERISTICS OF BUTYL-COATED FABRICS (RUNS 5 THRU 19)

Run No.	Fabric	Coating*	Breaking Strength (lb.) Warp	Breaking Strength (lb.) Fill	Elong. at Break % Warp	Elong. at Break % Fill	Tear (lb.) #5134 Warp	Tear (lb.) #5134 Fill	Mod. at 50% Elong. (lb.) Warp	Mod. at 50% Elong. (lb.) Fill	Adhesion (lb./2 in.)	Low Temp. Resist. #5874	Notes
5	Style #1	3/3	50	15	110	400	6.9	7.3	1.76	0.72	Inf.[1]	Passed 20°F	All samples cured in circulating air oven except where noted otherwise.
13	Style #[illegible]	1/1/1/1	51	15	85	330	-	-	2.1	0.53	0.5	"	
14	Style #5	3/1/1	54	24	80	280	-	-	5.6	0.98	Inf.	"	
14	Style #5	3/1/1/1	47	24	80	275	-	-	5.0	1.0	Inf.	"	
16	Style #4	3/2/2	70	23	100	290	10.0	7.7	3.5	1.6	Inf.	"	
16	Style #4	3/2	69	25	100	290	8.8	7.0	3.4	1.4	Inf.	"	
17	Style #4	3/2	69	25	100	290	8.9	7.2	3.4	1.5	Inf.	"	Cured in RF Vulc.
18	Style #5	2/2	75	22	75	310	13.7	10.6	6.8	1.1	4.5	"	
18	Style #5	2/1/1	80	22	75	315	13.0	10.7	5.5	1.4	4.8	"	
18	Style #5	3/2	83	23	70	365	11.0	12.0	5.2	1.2	4.3	"	
18	Style #5	3/1/1	70	24	70	320	13.7	10.7	5.1	1.1	5.2	"	
18	Style #2	3/2/1	53	22	85	300	7.4	6.8	6.0	1.4	5.2	"	
19	Style #2	3/1/1	50	24	110	250	8.4	7.5	2.9	1.4	Inf.	"	
19	Style #3	2/1/1	50	21	85	245	7.9	9.8	4.2	1.2	Inf.	"	
19	Style #1	3/1/1	20	13	160	470	6.5	7.0	1.30	1.0	5.0	"	
19	Style #1	2/2/1	20	13	170	490	5.9	6.9	1.70	1.0	Inf.	"	
19	Style #4	2/1/1	80	23	85	400	12.0	9.6	3.0	1.0	5.2	"	

Note: * Each figure represents the thickness of the coating in mils. The number of figures represents the number of coats. For example: 3/1/1 would be interpreted to mean a three-coat application, the first coat being 3 mils thick, the second, 1 mil thick and the third, 1 mil thick.

1. "Infinite" adhesion means that the film or fabric failed without delamination occurring.

TABLE [illegible]

[illegible]

Test Method:	Overall Wgt. 5041	Breaking Strgth. 5102		Elong. % 5102		Tear 5134		Mod. at 50% Elong.		Adhes. 5970	Low Temp. Resist. 5874	Hydro Static 5512	After Stretch of Coating 5972
Sample Size:	2x2	1x6		1x6		3x8		2x3		2x6	8x8	4x4	2x6
Sample No.	oz/yd²	Warp lb	Fill lb	Warp	Fill	Fill lb	Warp lb	Warp lb	Fill lb	lb/2in.		(Mullen)	
1	10.30	56	16	75	360	10.57	6.85	5.4	[illegible]	9.6	-52°C ok	[illegible]	36.7% coat bubbled
	10.15	54	12	75	350	11.38	6.56	5.4	1.75	8.4	-55°C ok	61	41.0% burst
	10.38	56	16	70	400	11.40	6.82	6.0	1.60	9.3	-52°C failed	58	45% burst
	10.57	52	16	70	365	10.57	6.12	10.0	[illegible]	[illegible]		55	41.5% burst
	9.72	52	13	70	340	9.38	6.32	10.5	1.7	8.7		70	44-bubbled 37.2%
Avg.	10.22	53.6	14.6	71	363	10.66	6.53	7.5	1.77	[illegible]		63	-41.4%
6	7.68	46	12	90	360	9.66	6.37	4.5	[illegible]	8.80		30	burst 33%
	7.44	40	12	65	340	8.73	6.52	4.7	1.34	8.52	-52°C ok	42	leak 30%
	7.10	53	15	75	380	11.78	6.74	5.0	1.30	8.35	-55°C ok	34	100%
	7.81	45	16	80	360	6.44	5.18	8.6	1.60	8.30	-59°C failed	57	100%
	7.14	58	15	75	380	5.62	6.56	9.2	1.70	9.25		36	100%
Avg.	7.43	48.4	14	77	364	3.65	5.47	6.4	1.48	8.44			
7	8.49	51	12	85	330	8.40	6.39	5.0	1.65	16.8	-52°C ok	70	burst-37%
	9.16	42	14	70	360	10.22	6.12	4.8	1.40	16.4	-55°C failed	68	burst-41.2%
	9.72	52	14	85	350	7.56	6.22	5.4	1.42	14.2		68	burst-38.3%
	9.29	49	16	70	390	8.72	[illegible]	8.4	1.85	[illegible]		75	burst-[illegible]
	9.81	59	16	80	360	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		54	burst-[illegible]
Avg.	9.29	51	14.4	76	358	8.71	[illegible]	[illegible]	1.61	[illegible]		57	burst-[illegible]
8	7.80	49	13	90	390	10.20	[illegible]	4.0	1.36	8.7	-52°C ok	42	31%
	8.51	47	14	65	390	9.87	[illegible]	4.4	[illegible]	[illegible]	-55°C ok	42	[illegible].5%
	8.12	45	15	80	400	10.48	6.42	4.4	[illegible]	[illegible]	failed	45	31%
	8.30	66	14	70	380	7.06	[illegible]	9.2	[illegible]	[illegible]		34	23.5%
	8.30	49	15	70	380	7.53	[illegible]	[illegible]	1.16	[illegible]		42	-42.0%
Avg.	8.21	51.2	14.2	79	388	9.02	[illegible]	6.6	1.44	8.4		35	[illegible]
9	8.34	42	12	90	310	6.50	7.83	[illegible]	2.6	9.86	-52°C ok	42	leak-57.3%
	8.08	37	17	70	360	[illegible]	[illegible]	3.4	[illegible]	[illegible]	-56°C failed	39	leak-[illegible]
	8.24	45	14	95	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		[illegible]	leak-[illegible]
	9.06	32	15	90	[illegible]	[illegible]	[illegible]	5.6	[illegible]	[illegible]		[illegible]	burst-[illegible]
	[illegible]	35	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		[illegible]	[illegible]
Avg.	8.74	38	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		[illegible]	[illegible]

TABLE V-[illegible]

MUSTARD AND GB PENETRATION OF SAMPLES FROM [illegible] NO. [illegible]

Sample No.	Time in Minutes for Penetration: Mustard HD-100 Minutes Required	Time in Minutes for Penetration: GB-200 Minutes Required
1 (10.2oz/yd²)	Four samples, [illegible]20 minutes each	[illegible]9, 35[illegible], [illegible], 389, 354, 379, 43[illegible]
6 (7.4oz/yd²)	Four samples, 40 minutes each	[illegible], [illegible], 8, 8, 25, 40, 55, 70
7 (9.3oz/yd²)	Four samples, 95 minutes each	130, 17[illegible], [illegible], 250, 250, 2[illegible], 2[illegible], [illegible]
8 (8.2oz/yd²)	Four samples, 60 minutes each	[illegible], 115, 115, 115, 125, 125, 1[illegible], 140
9 (8.7oz/yd²)	Four samples, 45 minutes each	85, [illegible], 85, 110, 110, 130, 130, 140

TABLE VI

DATA ON COATING RUNS 23-28

Cement Used: Butyl Comp. III, 33% in Xylol   Cure: 90'/300°F   Fabric Used: HC-24-A

Carter Rice 60 lb. Silicone Paper used as release paper

Run No.	Drying Temperatures °F 1st Zone	2nd Zone	3rd Zone	4th Zone	No. of Coats	Length of Fabric (Yd)	Aver. Width of Uncoated Fabric (in)	Aver. Width of Coated Fabric (in)	Notes
23	175	175	160	100	3	60	54	48	All samples cured in R.F. Vulcanizer -90' at 300°F.
	"	"	"	"	4	130	54	46	
	"	"	"	"	5	125	54	45	
	"	"	"	"	6	30	54	44	--------
24	195	200	175	100	2	10	18	16 1/2	Rubber-coated canvas duck fabric used to provide a semi-rigid substrate and prevent shrinkage of the HC-24A during processing. --------
25	175	175	160	100	4	10	18	18	First attempt at multiple coating of the release paper followed by a single transfer operation. --------
26	175	175	160	100	4	20	18	18	Scaled up version of Run 25. about 200 yds. of paper was coated to determine roll-up and unwind problems. --------
27	190	190	165	100	3	5	54	53	The 3-coat sample was spoiled because of water leakage on the film.
	190	190	170	100	4	5	54	53	The other two were submitted.
	190	190	170	100	5	5	54	53	
28	215	225	180	-	3	-	-	-	Run aborted because of film sticking to the reverse side of paper.

TABLE VII

PREPARATION OF PRODUCTION SAMPLES (RUNS 29 THROUGH 37)

Run No.	Coat No.	Belt Speed Ft/min.	Knife Setting (Mils)	Total Dry Film Thick- ness (Mils)	Drying Zone Temps. °F Zone 1	Zone 2	Zone 3	Zone 4	Ydge. Coated	Aver.Wt. of Coated Fabric	Notes
29	1	10	19	1.25	200	180	185	140			Part of three 100 yd.(three different coating wgts.) runs. This run was intended to represent 10 oz/yd² fabric.
	2	10	19	2.50	200	175	175	140	82	9.55	
	3	10	19	4.0	210	175	180	140			
	4	10	20	4.5	215	225	180	140			
30	1	7.0	16	0.50	215	180	180	140			Part of the three 100 yd. (3 coating wgt.) series. This run represented 8 oz/yd² material.
	2	10.0	17	2.0	"	"	"	"	122	7.62	
	3	10.0	18	3.0	"	"	"	"			
	4	10.0	19		"	"	"	"			
31	1	7.0	16	0.5	210	175	180	140			Part of the three 100 yd. (3 coating wgt.) series. This run represented the heaviest (nominally 11 oz/yd²) material.
	2	10.0	16	1.25	"	"	"	"			
	3	10.0	17	2.0	"	"	"	"			
	4	10.0	17	2.50	"	"	"	"			
	5	10.0	18	3.5	"	"	"	"	120	10.51	
	6	10.0	18	4.5	"	"	"	"			
	7	10.0	19	5.0	"	"	"	"			
	8	10.0	20	-	"	"	"	"			
32	1	7.0	15	0.5	215	180	180	140			This small section was intended as part of Run #29.
	2	10.0	16	1.0	"	"	"	"			
	3	10.0	17	2.0	"	"	"	"	17	9.85	
	4	10.0	17	3.0	"	"	"	"			
	5	10.0	18	4.0	"	"	"	"			
	6	10.0	19	-	"	"	"	"			
33	1	7.0	16	0.5	200	185	180	160			
	2	10.0	16	1.5	"	"	"	"			
	3	10.0	17	2.0	"	"	"	"			
	4	10.0	17	3.0	"	"	"	"	93	9.57	
	5	10.0	18	3.5	"	"	"	"			
	6	10.0	18	3.5	"	"	"	"			
	7	10.0	19	5.3	"	"	"	"			
	8	10.0	21	6.5	"	"	"	"			

TABLE VII
(Cont'd)
PREPARATION OF PRODUCTION SAMPLES (RUNS 29 THROUGH 37)

Run No.	Coat No.	Belt Speed Ft/min.	Knife Setting (Mils)	Total Dry Film Thick-ness (Mils)	Drying Zone Temps. °F Zone 1	Zone 2	Zone 3	Zone 4	Ydge. Coated	Aver. Wt. of Coated Fabric	Notes
34	1	7.0	16	0.3	200	185	180	140			
	2	10.0	16	1.0	"	"	"	"			
	3	10.0	17	2.0	"	"	"	"			
	4	10.0	17	2.7	"	"	"	"	141	11.19	
	5	10.0	18	3.7	"	"	"	"			
	7	10.0	21	13.5 (with cloth)			"	"			
	6	10.0	19	4.8	"	"	"	"			
	8	10.0	19	20.9		"	"	"			
35	1	7.0	16	0.5	200	185	180	160			
	2	10.0	16	1.5	"	"	"	"			
	3	10.0	17	2.4	"	"	"	"			
	4	10.0	17	3.0	"	"	"	"	(35A)		
	5	10.0	18	3.5	"	"	"	"	121	10.71	
	6	10.0	18	4.5	"	"	"	"	(35B)		
	7	10.0	19	5.5	"	"	"	"	77	11.07	
	8	10.0	21	6.5	"	"	"	"			
36	1	7.0	16	0.5	200	200	180	140			
	2	10.0	16	1.5	"	"	"	"			
	3	10.0	17	2.3	"	"	"	"			
	4	10.0	17	3.5	"	"	"	"			
	5	10.0	18	4.5	"	"	"	"	7.8	10.31	
	6	10.0	18	5.0	"	"	"	"			
	7	10.0	19	5.7	"	"	"	"			
	8	10.0	21	-	"	"	"	"			
37	1	7.0	16	0.5	200	180	180	140			
	2	10.0	16	1.5	"	"	"	"			
	3	10.0	17	2.3	"	"	"	"			
	4	10.0	17	3.0	"	"	"	"	141	10.54	
	5	10.0	17.5	3.5	"	"	"	"			
	6	10.0	18	4.7	"	"	"	"			
	7	10.0	19	5.5	"	"	"	"			
	8	10.0	21.5	-	"	"	"	"			

TABLE VIII

TEST DATA ON RUNS 23 THROUGH 37

Run No.	No. of Coats	Overall Wgt. (#5041) oz/yd^2	Breaking Strgth (#5102) lb Warp	Breaking Strgth (#5102) lb Fill	Elong. (#5102) % Warp	Elong. (#5102) % Fill	Tear (#5134) lb Warp	Tear (#5134) lb Fill	Mod. at 50% Elong. (lb/2 in) Warp	Mod. at 50% Elong. (lb/2 in) Fill	Adh. (#5970) (lb/2 in)	Low * Temp. Resist.	Mullen Hydro Static Resist. (#5512)	After-Strgth. of Coating (#5972)
23	3	7.64±0.3	44	26	146	238	8.19	9.94	2.4	1.6	6.5	-60°fld. -58° "	17	0
	4	8.66±0.6	57	20	98	280	8.03	8.67	6.6	1.9	8.0	-56° ok -58°fld. -36° ok -54° ok	40	17.0
	5	10.74±0.8	59	20	80	266	7.17	5.28	8.0	2.8	11.3	-60° fld. -58° fld. -56° ok -55° ok	61	34.4
	6	12.42±0.8	-	-	-	-	-	-	-	-	-	-	-	-
24	This run was aborted because of excessive creasing of the fabric.													
25	4	11.2±0.4	Very short run-no testing of product.											
27	3	8.5±0.4	41	34	170	205	-	-	-	-	-	-	leak at press.	
	4	9.6±0.5	57	32	155	190	-	-	-	-	-	-		
	5	11.1±0.5	59	35	160	185	8.5	8.7	7.5	2.6	8.5	-60°fld. -56° ok -59° ok	68	39
28	Aborted because of mechanical difficulties													
29 and 32	4	9.55±0.4	41	31	180	210	8.47	8.55	2.36	2.52	-	-58°fld. -56° " -54° ok		
	6	9.85±0.4	This roll (17 yds) was sent as part of Run #29 to make up for short yardage.											

*All temperatures °C.

TABLE VIII
(Cont'd)
TEST DATA ON RUNS 23 THROUGH 37

Run No.	No. of Coats	Overall Wgt. (#5141) oz/yd^2	Breaking Strgth. (#5102) lb Warp	Breaking Strgth. (#5102) lb Fill	Elong. (#5102) % Warp	Elong. (#5102) % Fill	Tear (#5134) lb Warp	Tear (#5134) lb Fill	Mod. at 50% Elong. (lb/2 in) Warp	Mod. at 50% Elong. (lb/2 in) Fill	Adh. (#5970) (lb/2 in)	Low Temp.* Resist.	Mullen Hydro-Static Resist. (#5512)	After-Strgth. of Coating (#5972)
30	4	7.62 ± 0.5	44	27.7	190	270	8.24	8.80	2.26	2.52	8.6	-57°fld. -55° ok -54° ok	9.0	0
31	8	10.51 ± 0.5	41	32	195	200	7.49	8.04	2.36	1.92	4.0 to inf	-59°fld. -56°fld. -54° ok	35	17.5
33	6	9.57 ± 0.4	35.5	30.5	165	230	8.50	7.64	2.11	1.65	2.0 to inf	-59°fld. -56° ok	38	16.0
34	8	11.19 ± 0.5	43	35	170	190	7.5	8.5	3.7	2.9	4.0 to inf	-60°fld. -58° " -56° ok	52	50
35	8	10.9 ± 0.6	41.0	32	190	230	9.82	8.15	2.61	2.33	4.0 to inf	-53°fld. -57° ok	28	27
36	8	10.31 ± 0.4	42.0	31.5	180	200	9.5	8.7	2.88	2.25	6.0 to inf	-59°fld. -56° ok	32	30
37	8	10.64 ± 0.4	37.0	33.0	185	210	7.33	9.13	2.43	1.93	2.0 to inf	-58°fld. -56° ok	33	33

* All Temperatures $^{\circ}$C.

TABLE VIII "A"

MUSTARD H AND BG PENETRATION DATA ON RUNS 23 THROUGH 37

Run No.	No. of Coats	Overall Wgt. (#5041) oz/yd^2	MUSTARD H (100 min. required) (10 Determinations Each)	BG ([illegible] min. required) (10 Determinations Each)
23	3	7.6±0.3	All 26 minutes	[illegible], 45, [illegible], 45, [illegible], 60, 65, 63, [illegible], 60
	4	8.7±0.6	36, 36, 46, 46, 36, 46, 36, 46, 46, 46	65, 65, 110, 110, 30, 30, 28, 52, 28, 60
	5	10.7±0.8	60, 60, 50, 50, 55, 55, 50, 50, 55, 55	138, 138, 158, [illegible], 158, [illegible], 138, 138, 138, 138
	6	12.4±0.8	All 95 minutes	All 240 minutes.
27	3	8.5±0.4	Not tested (too many "pin holes" evident on inspection)	
	4	9.6±0.5	Not tested (a few "pin holes" noted on inspection)	
	5	11.1±0.5	All 90 minutes	251, 270, 270, 250, 345 (5 determinations only)
34*	8	11.2±0.5		
35*	8	10.9±0.6		
36*	8	10.3±0.4		
37*	8	10.6±0.4		

Note: * Runs 34-37 represent 500-yard product run.

APPENDIX

Identification of Abbreviations and Trade Names

Trade Designation	Composition	Manufacturer
Ceresin Wax	Ceresin wax, natural or synthetic	Akron Chemical Co.
Dixie Clay	Kaolin, hard clay	R. T. Vanderbilt Co., Inc.
MBTS (Accelerator)	Benzothiazyl disulfide	American Cyanamid
Pelletex	Carbon black in pelletized form	Godfrey L. Cabot Inc.
Pennac TM-526	Mixed (methyl and ethyl) tetraalkyl thiuram disulfide	Pennsalt Co.
Sulfasan R	4, 4' Dithiomorpholine	Monsanto Chemical Co.
531 Yellow Powder	Organic Pigments	Disco Co.
547 Green Powder	Organic Pigments	Disco Co.
507 Red Powder	Organic Pigments	Disco Co.
Stripkote AR	Release paper, silicone coated, one side only	S. D. Warren, Co.
TiO_2	Titanium Dioxide Anatase Type A168-LO	Titanium Pigment Co.
Zinc Oxide	French process Zinc Oxide Kadox 15	New Jersey Zinc Co.
Sulfur	Refined natural Sulfur RM 99	HM Royal Inc.
Diethylene Glycol	Diethylene Glycol	Allied Chemical Corp.
Zinc Stearate	Zinc Stearate	H & S Chemical Co.

Unclassified
Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author)
Rhee Division
Rohm and Haas Company
Warren, Rhode Island

2a. REPORT SECURITY CLASSIFICATION
Unclassified

2b. GROUP

3. REPORT TITLE
Development of a Lightweight Butyl-Coated Stretch Fabric

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)
Final Report for period: Oct. 6, 1966 through April 6, 1968

5. AUTHOR(S) (First name, middle initial, last name)
James P. Shelley

6. REPORT DATE
April 1968

7a. TOTAL NO. OF PAGES
29

7b. NO. OF REFS

8a. CONTRACT OR GRANT NO.
DAAG 17-67-C-~~009~~ 00...

b. PROJECT NO.
1J025601A032

c.

d.

9a. ORIGINATOR'S REPORT NUMBER(S)

9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)
68-63-CM: C&OM-48

10. DISTRIBUTION STATEMENT
This document has been approved for public release and sale; its distribution is unlimited.

11. SUPPLEMENTARY NOTES

12. SPONSORING MILITARY ACTIVITY
U.S. Army Natick Laboratories
Natick, Massachusetts

13. ABSTRACT

This study was directed toward the development of a technique for the application of multiple thin layers of butyl rubber on a stretch nylon substrate. Since the films of butyl, being of the order of about 1 mil each in thickness, were too thin to calender, a transfer coating technique was considered the best approach, although some consideration was given to the possibility of direct coating. Transfer coating was accomplished by knife coating a thin film of butyl rubber from a (33% solids) cement on to release paper, drying the film, and then transferring the film from the paper to the fabric by passing film and fabric between doubler rolls. Several problems were encountered with this method but the most serious was extensive shrinkage and loss of warp stretch in the fabric which resulted largely from excessive handling. Several variations in the process designed to minimize fabric handling were explored. The present, and most successful, of these may be described briefly as follows:

Instead of each coat being transferred individually to the fabric, all of the coats are applied to the paper. The fabric is applied on top of the last coat (while the film is still wet) and the entire combination of fabric, film and paper is subjected to the vulcanization process. The release paper is peeled off the coated fabric after vulcanization.

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Unclassified
Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Transferring	8					
Applying	8					
Vulcanization	8					
Stretch fabrics	1		9			
Butyl rubber	1					
Rubber coatings	1		8			
Coated fabrics	2					
Protective clothing	4		4			